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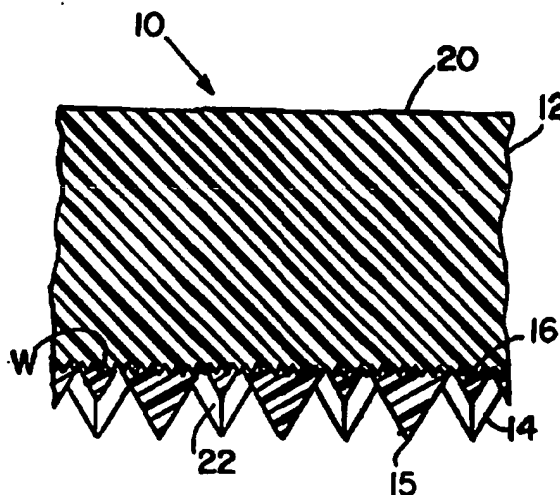
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(54) Title: RETROREFLECTIVE PRISM ARRAYS WITH UNIFORM LIGHT DISTRIBUTION

(57) Abstract

Retroreflective sheeting and a method of forming and using such sheeting is described in which a base body is formed with closely spaced microprisms. The microprisms have window facets extending across the body portion in a plane with retroreflecting facets extending from the window facets to apices. Either the base body surfaces or the side facets are textured to deviate the path of light entering and/or retroreflected from the prisms to provide a uniform distribution of light.



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RETROREFLECTIVE PRISM ARRAYS WITH
UNIFORM LIGHT DISTRIBUTION

Background of the Invention

Conventional retroreflecting prism or corner cube
5 arrays of the type described in U.S. Patent No. 5,171,624
(incorporated herein by reference in its entirety) create a
diffraction pattern having a characteristic void outside of
a central maxima and a six lobed light energy distribution,
as shown in FIG. 3, herein, taken from FIG. 5 of the
10 referenced patent. The six lobes of energy formed about
the central maxima represent the energy cones of the first
order of diffraction. Such an energy distribution is
undesirable because of the high degree of variation of
energy level throughout the retroreflected beam.

15 Summary of the Invention

In accordance with the present invention, uniform
distribution of light energy from retroreflective sheeting
is achieved by texturing a surface of the sheeting which is
in the path of the light energy. The retroreflective
20 sheeting is formed of a base body with microprisms having a
window and three side surfaces. The microprisms extend
across the body in a plane adjacent the base body surface.
Preferably, the texturing is formed on a base body surface,
in which case the base body should have an index of
25 refraction n_1 which is different than the index of
refraction n_2 of the retroreflective microprisms. The
preferred texture may range from macro-size (peak to valley
height (H) in the order of .0002 inch to .0008 inch)
undulations on an otherwise smooth or glossy surface
30 (hereinafter macrotexture) to a combination of macrotexture

-2-

and micro-size grooves having a peak to valley height (h) in the order of 0.00002 inch to 0.00004 inch. The texturing causes light retroreflected from the prism facets to be deviated slightly, resulting in a more uniform energy distribution of the retroreflected light and wider observation angle performance.

Alternatively, the prism facets, per se, may be textured using a chemical etching process, with a similar result.

10 Brief Description of the Drawings

FIG. 1 is a plan view of a portion of a back surface of retroreflective sheeting made in accordance with the invention.

FIG. 2 is an enlarged sectional view of the sheeting 10 of FIG. 1 taken along lines A-A.

FIG. 3 is a schematic drawing of a typical retroreflected energy pattern generated by prior art microprism material when the prisms are about 0.006 inch on centers.

FIG. 4 is a schematic drawing of a retroreflected energy pattern generated by the retroreflective sheeting of the present invention.

FIG. 5 is an enlarged partial sectional view of a base body 12 having a combination of micro and macro texturing.

FIG. 6 is an enlarged view as in FIG. 5 showing only macro-texturing.

FIG. 7 is a plot of light intensity versus observation angle for three samples.

FIG. 8 is an enlarged sectional view of sheeting of an alternate embodiment.

FIG. 9 is a plan view of an alternate embodiment of the invention wherein the texturing is formed on the prism facets.

-3-

FIG. 10 is an enlarged view of a single prism of FIG. 9.

Detailed Description of the Invention

Turning now to the drawings and, in particular, FIGS. 1-4, the invention will now be described in detail in connection therewith.

FIG. 1 is a plan view of the retroreflective prism sheeting of the present invention taken from the bottom of the sheeting 10. Note: The term "sheeting" as used herein refers to relatively thin sheet-like structures, as well as thicker members, laminates and the like, which have a substantially planar front face upon which light rays impinge upon a body portion 12 which is substantially transparent to the light rays. The term "microprisms" refers to closely packed or spaced prisms extending in a plane spaced from and generally parallel to and opposite the front face 20 of body 12. The microprisms have a spacing between their apices 15 in the order of 0.002-0.0025 inches and each is formed with three side surfaces or side facets S1, S2 and S3 disposed along three intersecting planes and a window facet W.

In the method for producing the microprism reflective sheeting 10 of the present invention, a mold is formed with closely spaced microprism cavities therein. A synthetic prism forming transparent resin material is formed on the mold to fill the cavities to form microprisms therein and to provide a continuous body across the surface of the mold. A web of body material 10, having an index of refraction n_1 which differs from the index of refraction n_2 of the microprisms 14 is applied to the microprisms in the mold and adheres thereto as the composite structure is removed from the mold.

In accordance with a first embodiment of the invention, the bottom, or inner portion, of the base body 12 has a matté finish formed of a composite micro and macro

-4-

surface texture 16 embossed, etched, or otherwise formed thereon. Body 12 is preferably formed of a vinyl film. The outer surface 20 is formed with a smooth glossy finish. The micro/macro texture 16 on the bottom of the film is

5 sufficient to create scattering of the incident and the retroreflected light resulting in a uniform light intensity retroreflected pattern, as shown in FIG. 4. This scattering is very beneficial since it causes a very uniform beam of highly divergent retroreflected light to be

10 created with greatly improved wide observation angle light distribution. This is illustrated by the prior art pattern shown in FIG. 3, which as previously noted, consists of a maximum central lobe of light 36 surrounded by six lobes of first order diffracted light cones 30 with little or no

15 light formed in the areas between the lobes and the central maxima.

In FIG. 4, the shaded area illustrates the light pattern formed by the present invention, which is substantially uniform over the entire area. The previous

20 lobes of FIG. 3 are shown in dotted lines and are substantially indistinct in the pattern as formed.

It is important that the refractive index of the materials used for the base body 12 and the material used in the microprisms 14 differ. Preferably, a hard acrylated

25 epoxy material (n_2 equal to about 1.49) is used for the prism structure 14. A 0.10 inch thick vinyl clear film (n_1 equal to about 1.52) with micro and macro surface texture 16 on the inner side may be used for the body material 12.

The overall uniform light distribution maintains its

30 uniformity as the sheeting 10 is rotated to increase the angle of incidence between the incoming light beam and a sample surface, as measured using a laser beam operating at a wavelength of 632.8 nanometers. At an angle of incidence of 45° , the light pattern is still very uniform but is

35 slightly elliptical in shape as a result of the elliptical

-5-

shape of the laser beam as it intersects with the rotated sample and the change in shape of the hexagonal effective aperture of the retroreflecting prisms.

Two examples of the type of films which can be used
5 for the textured body 12 of the invention are illustrated in FIGs. 5 and 6. Film A is formed of textured vinyl with a matté finish embossed on the outer surface during calendering of the vinyl. The film can be purchased from Renolit Corporation (Germany) under the tradename "Renolit
10 Matte 160/2."

The Film A of FIG. 5 has a random or pseudo-random macro texture of about 0.015 to 0.025 inches with a random or pseudo random micro-texture 16 imposed thereon having a peak to valley high (h) in the order of 0.00002 to 0.00004
15 inches; whereas the peak to valley height H1 of the macro-texture is in the order of 0.0002 to 0.0008 inches.

The film B of FIG. 6 is an alternate choice of a textured film for use as the textured body 12 of the invention. Film B is provided with a random or pseudo-
20 random macro-textured surface texture formed of vinyl material and sold by Renolit Corporation under the tradename Orang 4. The macrotexture 16' has a periodicity P2 of about 0.050 inch to 0.070 inch and a peak to valley height H2 in the order of 0.0002 to 0.0008 inch with no
25 microtexture.

The improvement in observation angle performance of the retroreflective sheeting of the present invention over standard retroreflective sheeting is illustrated in the plot of FIG. 7. Three curves of Brightness (cd/lux/m^2)
30 versus Observation Angle (degrees) are plotted in FIG. 7. Curve A shows the results for sheeting made from Film A, Curve B shows the results for similar sheeting with the Film B body substituted and Curve C shows the results for a similar test using non-textured standard film.

-6-

As may be seen the light intensity for the prior art standard film decreases rapidly between 0.33 degrees and 0.5 degrees, whereas the fall off in intensity is more gradual with film B and even less steep with film A.

5 The brightness of the central area 36' of FIG. 4 may be significantly increased by applying a metallic reflective coating, such as a coating of 600 Å of aluminum 40 to the facets 22' of the prism structure 14' (as shown in FIG. 8). The surrounding area still remains very bright
10 and in this alternative embodiment, no visible secondary maxima 30' remain. The micro/macro texture 16 on the body portion 12 creates a greater divergence of the light. The divergence is 1.42° for a sample formed of .0054 inch pitch, 3° tilt prisms made from acrylated epoxy resin
15 applied to the textured side of a .010 inch vinyl body portion 12. In comparison, a standard .0054 pitch 3° tilt retroreflective sheeting formed with a .010 inch vinyl body portion with an untextured glossy bottom surface has a divergence of 1.12°. Note that it is not necessary to have
20 the textured side of the body portion 12 located at the interface between the body portion and the microprisms 14. The same scattering effect can be produced by having the textured finish 16' on the outside surface 20 of the body portion 12, as shown in FIG. 8. However, the amplitude of
25 the texture must be much lower. An amplitude which is 1% to 8% of the macro texture amplitude in FIGS. 5 and 6 is preferred.

In an alternate embodiment, shown in FIGS. 9 and 10, the prism facets S1, S2 and S3 are textured instead of the
30 bottom surface of the base body, with a similar result. The texturing of the facets causes light retroreflected from the facets to be deviated slightly resulting in uniform energy distribution of the retroreflected light.

Texturing of the facets can be accomplished by
35 chemical etching, in a random fashion, the prism faces of a

7

-7-

plastic (i.e., butyrate) mold used to form the prisms. One way of doing this is to form a granulated mold surface using the process described in U.S. Patent 3,718,078 to Plummer and incorporated in its entirety herein by
5 reference.

Yet another preferred approach for texturing the prism facets involves applying a spin coat photoresist application to a nickel mold of the retroreflective sheeting. The resist is then exposed to light from a
10 scanning laser beam projected through a diffuse screen to form a speckled pattern in the resist. The resist is then developed, for example, by a UV bake, and then etched in the exposed areas of the photoresist. The remaining resist is removed leaving a speckled imprint on the mold which can
15 be transferred to the prism facets formed in the mold. The depth of the surface texture, so formed, is about one millionth of an inch and the "crater" width of the "speckles" is about .0011 inches.

Various synthetic resins may be employed for the cube-
20 corner formulations and for the sheet material including polymers of (alk) acrylic acid esters, such as polymethyl methacrylate and polybutyl acrylate; cellulose esters, such as cellulose acetate polymer, cellulose acetate/butyrate copolymer, and cellulose propionate polymer; vinyl halides,
25 such as polyvinyl fluoride; vinylidene halides, such as polyvinylidene chloride; monovinylidene aromatic hydrocarbon polymers, such as polystyrene and styrene/acrylonitrile copolymers; ethylenically unsaturated nitriles, such as polyacrylonitrile; polycarbonates;
30 polyesters, such as polyethylene terephthalate; polyphenylene oxide; polysulfones; and polyolefins, such as polyethylene and polypropylene. Interpolymers of various of the several above-mentioned types of monomers, e.g., vinyl chloride/vinyl acetate copolymers, may also be
35 employed, as may be mixtures of polymers. The particular

-8-

resin formulations selected for the components of the composite structure will vary depending upon the application, the thickness desired for the body member, the desire for flexibility, and the need for achieving interadhesion therebetween. For outdoor applications, materials which are moisture resistant, ultraviolet resistant and abrasion resistant are particularly advantageously employed at least for the body portion, since that portion is generally exposed to the atmosphere and requires good weathering characteristics. Moreover, it will be appreciated that body portion 12 may itself be a laminate of films or sheets of two different synthetic resins, and it may be provided with coatings of appropriate materials subject to the requirement that the mean index of refraction, i.e., composite index, of the composition structure should differ from that of the cube corner prism structure.

The resins preferably employed for the body portion include polyvinyl halide, polyethylene terephthalate, polyvinylidene chloride, polycarbonates, polysulfones and cellulose ester polymers. The resins preferably employed for the cube-corner formations comprise (alk) acrylic acid ester resins, acrylic-modified vinyl chloride resins, vinyl chloride/vinyl acetate copolymers, ethylenically unsaturated nitrile resins, monovinylidene aromatic hydrocarbon resins, olefin resins, cellulose ester resins, polysulfone resins, polyphenylene oxide resins and polycarbonates. Subject to the differing index of refraction requirement exemplary combinations for the body portion/cube-corner formations include polyvinyl chloride/acrylic modified polyvinyl chloride; polyvinyl fluoride/polyvinyl chloride; polycarbonate/polycarbonate; polyvinyl chloride/polymethyl methacrylate; polysulfon /polymethyl methacrylate; polysulfone/polyvinyl

-9-

chloride; and polyethylene terphthalate/polymethyl methacrylate.

In selecting the molding materials employed for the present invention, it should be remembered that long
5 lasting properties will require resins which do not have readily volatilizable plasticizers or other components, and which have an acceptable level of light stability. Thus, stabilized formulations are desirably employed when the resin itself is susceptible to light or oxygen degradation.
10 By proper selection of the resin systems, the sheet material may also provide a valuable degree of protection for the resin of the cube-corner formations, which may exhibit relatively poor stability when the cube-corner formations are reflectively coated and further coated with
15 a lacquer and/or adhesive. These coatings also may act as protective layers, since the body portions will, in many applications, serve as a barrier layer for ultraviolet radiation, vapor, gasses, etc.

It should be appreciated that the selection of
20 different resins for the two component portions, i.e., body 12 and prisms 14 of the product must recognize the need for compatibility of the two resins. For example, one resin must not contain a substance which is deleterious to the other and which will migrate thereinto across the
25 interface. Moreover, when plasticized materials are employed, it is desirable to use plasticizers which do not readily migrate, and/or to select formulations for both component resins in which the plasticizer contents are balanced so as to avoid a degree of migration therebetween
30 which might materially affect the properties of the component portions.

Although the cube-corner formations 14 in the illustrated embodiment have a uniform orientation within the array, it is possible to employ a pattern in which
35 certain of the cube-corner formations are disposed in such

-10-

a manner that their faces are not parallel to any of the faces of the adjacent cube-corner formations. Moreover, certain of the cube-corner formations may be disposed with their apices aligned other than vertically over the center
5 of their respective bases.

Equivalents

Those skilled in the art will recognize, or be able to ascertain, using not more than routine experimentation, many equivalents to specific embodiments of the invention
10 described specifically herein. Such equivalents are intended to be encompassed in the scope of the following claims.

-11-

CLAIMS

What is claimed is:

1. Retroreflective sheeting for retroreflecting light
comprised of:
 - 5 a) a body having a composite index of refraction n_1
and inner and outer surfaces extending in a plane
adjacent each other; one of said surfaces being
textured; and
 - 10 b) closely spaced microprisms having a composite
index of refraction n_2 ; which is different than
 n_1 , the microprisms extending across the body
portion in a plane adjacent the plane of the body
surfaces; and wherein the textured surface of the
15 body portion causes light retroreflected from the
microprisms to be deviated slightly resulting in
a uniform energy distribution of the
retroreflected light.
2. The sheeting of Claim 1 wherein the texture is formed
on the inner surface at an interface with the
20 microprisms.
3. The sheeting of Claim 2 wherein the microprisms have a
window facet disposed at the interface.
4. The sheeting of Claim 1 wherein n_1 and n_2 are in the
range of 1.4 to 1.7.
- 25 5. The sheeting of Claim 1 wherein the texturing
comprises a matté finish formed on one of the
surfaces.

-12-

6. Retroreflective sheeting comprised of:
- a) a base body portion having an outer planar surface and an inner surface;
 - b) closely spaced microprisms the microprisms having window facets extending across the body portion in a plane, adjacent the plane of the outer surface, with side facets extending from the window facets to apices and wherein light which propagates through the body portion enters the window facets and is retroreflected therefrom by the side facets; and
 - c) a textured surface in the path of the light to deviate the path and provide a more uniform distribution of retroreflected light.
7. The sheeting of Claim 6 wherein the textured surface is formed on one of the base body surfaces.
8. The sheeting of Claim 6 wherein the texturing is comprised of a combination of macroscopic undulations and microscopic grooves.
9. The sheeting of Claim 6 wherein the body portion has an index of refraction n_1 and the microprisms have an index of refraction n_2 and n_1 and n_2 are in the range of 1.4 to 1.7.
10. The sheeting of Claim 6 wherein the texturing is random.
11. The method of forming retroreflective sheeting comprised of the steps of:
- a) forming a body portion having an index of refraction n_1 with outer and inner surfaces;

-13-

- b) attaching closely spaced microprisms having window surfaces and side surfaces and an index of refraction n_2 ; which differs from n_1 , across the inner surface of the body portion;
- 5 c) texturing at least one of said surfaces; and wherein the textured surface causes light passing through the body portion and retroreflected from the microprisms to be deviated resulting in a more uniform energy distribution of the
- 10 retroreflected light.
12. The method of Claim 11 in which the textured surface is formed on a body portion surface.
13. The method of Claim 12 in which the textured surface is formed of microgrooves and macro undulations.
- 15 14. The method of Claim 12 in which the texturing is provided by embossing one side of a sheet of vinyl material.
15. The method of Claim 11 including the step of forming metallic reflective surfaces over the microprism side
- 20 surfaces.
16. The method of Claim 11 wherein the texturing comprises macro-size undulations.
17. The method of Claim 11 wherein the texturing comprises micro-size grooves.
- 25 18. Retroreflective sheeting comprised of:
- a) a base body portion having an outer planar surface and an inner surface;

-14-

- 5 b) closely spaced microprisms having window facets
 extending across the body portion in a plane,
 spaced from the plane of the outer surface, with
 retroreflecting facets extending from the window
 facets to apices and texturing formed on the
 retroreflecting facets for deviating the path of
 light retroreflected therefrom to provide a
 uniform distribution of the retroreflected light.
- 10 19. The sheeting of Claim 18 wherein the texturing is
 provided by chemical etch of molds forming the prisms.
20. The sheeting of Claim 18 wherein the texturing is in
 the form of a random granulated surface.
- 15 21. The method of forming retroreflective sheeting
 comprised of the steps of:
- a) forming a body portion n1 with outer and inner
 surfaces;
- b) attaching closely spaced microprisms having
 inclined facet faces;
- 20 c) texturing at least said faces; and wherein the
 textured faces cause light retroreflected from
 the microprisms to be deviated slightly resulting
 in a uniform energy distribution of the
 retroreflected light.
- 25 22. The method of retroreflecting light to produce a
 uniform energy distribution of the reflected light
 comprising the steps of:
- a) directing the light onto retroreflective sheeting
 formed of an array of closely packed microprisms
 having window and side facets; and

-15-

- b) deviating the path of said light entering and retroreflected from the side facets to produce said uniform energy distribution.

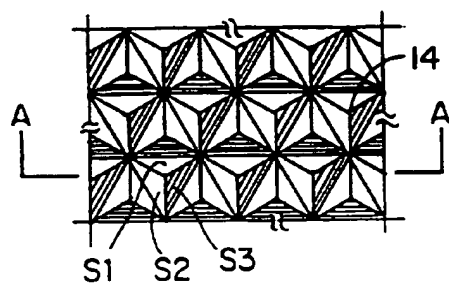


FIG. 1

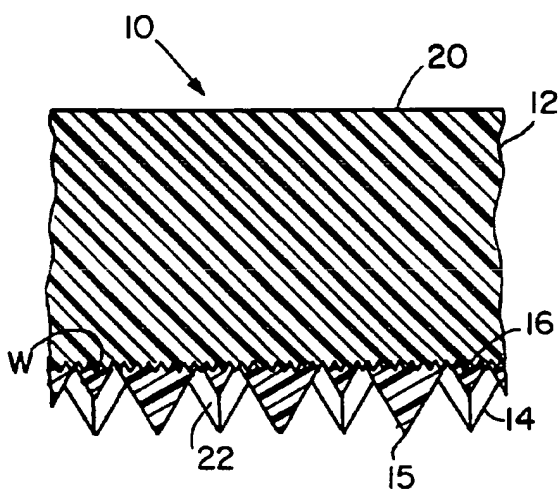


FIG. 2

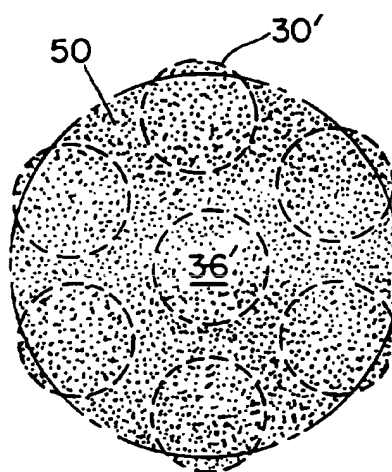


FIG. 4

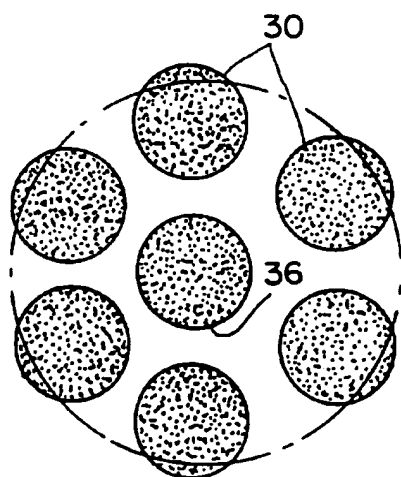


FIG. 3
PRIOR ART

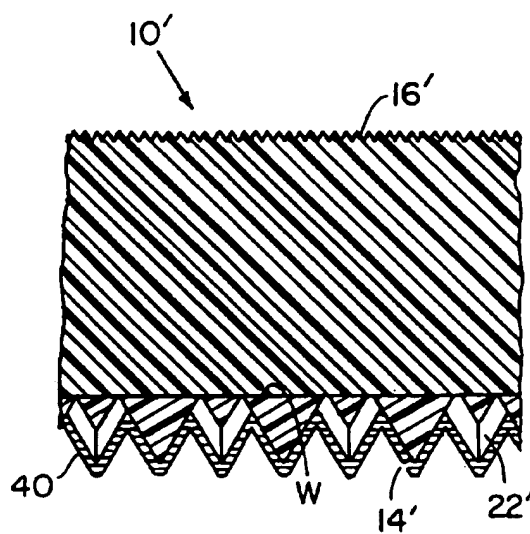


FIG. 8

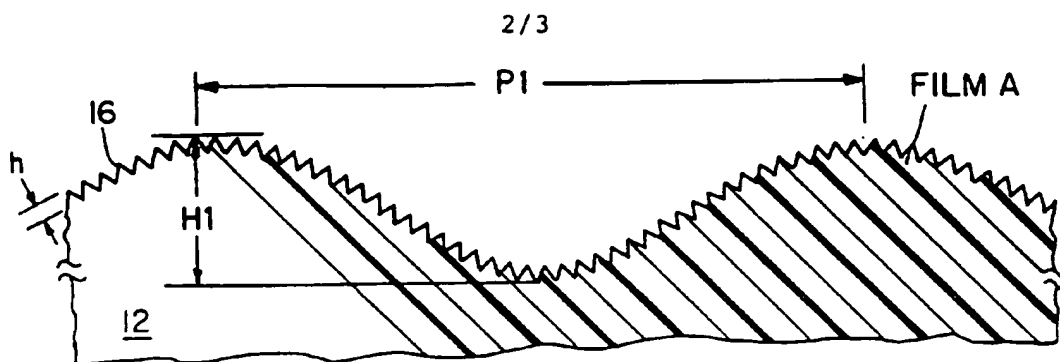


FIG. 5

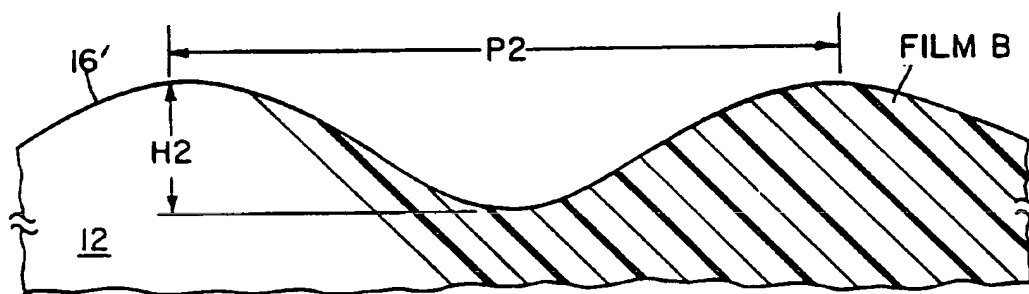


FIG. 6

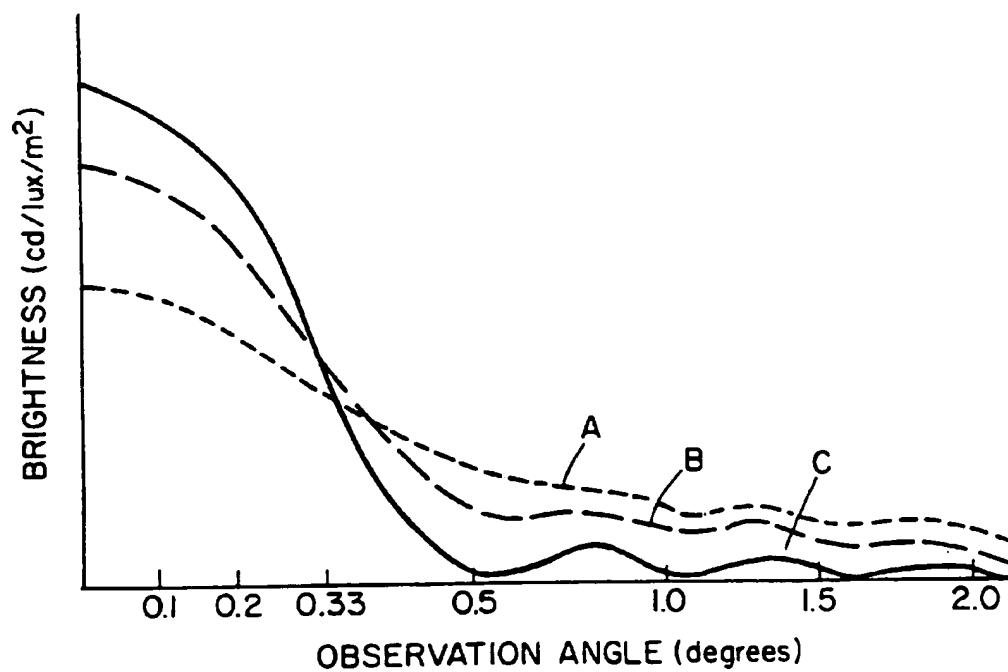


FIG. 7

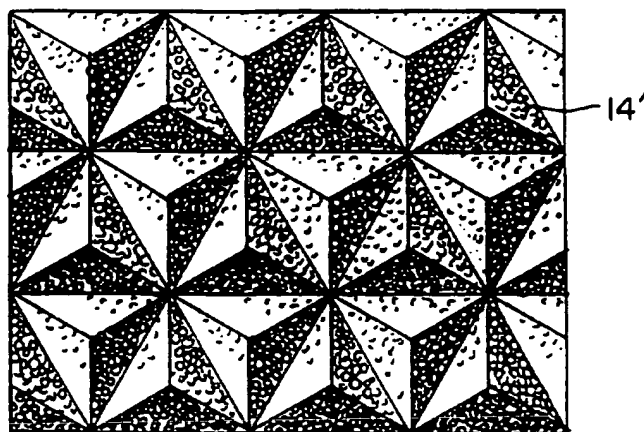


FIG. 9

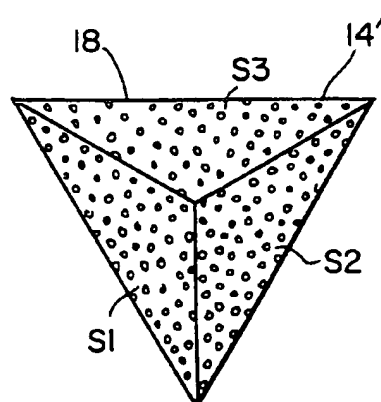


FIG. 10

INTERNATIONAL SEARCH REPORT

Int. Application No

PCT/US 96/01974

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 G02B5/124

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 G02B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,5 171 624 (H. WALTER) 15 December 1992 cited in the application see the whole document ---	1-22
A	WO,A,95 03558 (REFLEXITE CORP.) 2 February 1995 see the whole document -----	1-22

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

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Date of mailing of the international search report

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Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Sarneel, A

INTERNATIONAL SEARCH REPORT

Int. Patent Application No
PCT/US 96/01974

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-5171624	15-12-92	CA-A, C 2040909 DE-A- 4117911 FR-A- 2662819 GB-A, B 2245985 JP-A- 4232903	02-12-91 05-12-91 06-12-91 15-01-92 21-08-92
WO-A-9503558	02-02-95	US-A- 5491586	13-02-96